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**The Crystal Structure of *trans*(NO₂, N = C—CH₃)-(N-ethylidene-
-ethylenediamine)glycylglycinatonitrocobalt(III) Monohydrate,
[Co(glygly)NO₂(CH₃CH=en)]·H₂O***

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The crystal structure of *trans*(NO₂, N = C—CH₃)-(N-ethylideneethylenediamine)glycylglycinatonitrocobalt(III) monohydrate has been determined from 2519 diffractometric data. The crystals are monoclinic, space group *Pc*, with $a = 788.4(2)$, $b = 1514.9(2)$, $c = 1220.6(2)$ pm, $\beta = 120.82(4)$, $M = 339.19$, $Z = 2$, $D_m = 1790$, $D_x = 1800$ kg m⁻³. The structure was solved by the heavy-atom method and refined by the full-matrix least-squares technique to a final $R = 0.057$. The crystal structure consists of enantiomeric pairs of complex molecules and molecules of water. There are two crystallographically independent but chemically equivalent complex molecules, which differ only in conformation. The coordination around cobalt atoms is distorted octahedral. The glycylglycinato ligand is tridentate and bonded to the metal atom through carboxylato oxygen, amino nitrogen and peptide nitrogen atoms, forming two fused chelate rings, which are equatorially disposed. The diamine ligand is bidentate and contains the N-ethylidene group in the *trans*-position with respect to the nitro group. The methyl group of the aldehyde residue is in the *syn*-position with respect to the ethylenediamine residue.

INTRODUCTION

In the reaction of ethylenediamineglycylglycinatonitrocobalt(III) with acetaldehyde in aqueous solution at pH 11, four condensation products have been obtained.¹ One of them, (N-ethylideneethylenediamine)-glycylglycinatonitrocobalt(III), [Co(glygly)NO₂(CH₃CH=en)], appears as the result of the condensation of the aldehyde group and one of amino groups of the diamine ligand. On the basis of the data obtained from electronics, ¹H and ¹³C NMR spectra and paper chromatography, no conclusions could be made as to which amino group of the diamine ligand is involved in the condensation reaction *i. e.* *trans* or *cis* to NO₂. Therefore, an X-ray crystal structure analysis of the title compound was undertaken.

EXPERIMENTAL

Crystal data: 2 { [Co (glygly) NO₂ (CH₃CH = en)] · H₂O }, 2 [CoC₈H₁₆N₅O₅ · H₂O], $M = 339.19$, $a = 788.4(2)$, $b = 1514.9(2)$, $c = 1220.6(7)$ pm, $\beta = 120.82(4)^\circ$, $Z = 2$, $D_m = 1790$ $D_x = 1800$ kg m⁻³, space group *Pc*.

* Dedicated to Professor D. Grdenić on occasion of his 65th birthday.

The unit cell dimensions were initially obtained from rotation and Weissenberg photographs and later adjusted by least-squares refinement of the setting angles of 15 reflections centered on a Syntex P1 four-circle diffractometer, using Mo-K α radiation. The extinction of *h*0*l* reflections when *l* odd is consistent with space groups *Pc* and *P2/c*. The Patterson synthesis indicated space group *Pc*, which was confirmed by a successful refinement. The intensity data were collected on a Syntex P1 four-circle diffractometer, using graphite-monochromatized Mo-K α radiation and an ω -scan procedure. All independent reflections in the sphere $\theta < 30^\circ$ were measured; 2519 observations satisfying the criterion $I > 3\sigma(I)$ were used in the analysis. The intensity of a standard reflection, measured every 30 reflections, remained essentially constant throughout the data collection. The values of *I* and $\sigma(I)$ were corrected for Lorentz and polarization effects, but not for absorption nor extinction.

The structure was solved by a routine application of the heavy atom method; the atomic parameters were refined by full-matrix least-squares calculations. The function minimized was $\sum \omega \Delta^2$, where $\omega = 1/\sigma^2(I)$ and $\Delta = |F_o| - |F_c|$. All atoms were assumed to be uncharged. Values for the atomic scattering factors and anomalous terms for cobalt were taken from International Tables for Crystallography.²

Refinement of positional and thermal parameters (anisotropic for cobalt, carboxylato and water oxygen and nitrogen atoms, isotropic for the remaining nonhydrogen atoms), a total of 238 variables for 40 atoms in the asymmetric unit, terminated at *R* = 0.057, *R*_w = 0.073. In the final difference Fourier map a number of peaks were found in the positions which correspond to hydrogen atoms of the complex molecule, but they were not included in structure factor calculations. The final atomic parameters for nonhydrogen atoms, derived from the last cycle of least-squares refinement, are given in Table I, along with standard deviations estimated from the inverse matrix.

The main computer programs used on the CDC computer were FORDAP, Zalkin's³ Fourier program, NUCLS, a modification by J. A. Ibers and R. J. Doedens of the full-matrix least-squares program ORFLS,⁴ GEOM, K. W. Muir and P. Mallinson's program for molecular geometry and standard deviation and RING, written by L. K. Parkanyi, for ring conformation.

RESULTS AND DISCUSSION

The structure consists of two crystallographically independent enantiomeric pairs of the complex molecule and molecules of water, joined together by hydrogen bonds. A perspective drawing of the molecules with a numbering scheme of the atoms is given in Figure 1. Figure 2. represents an *a*-axis projection of the structure showing the packing and hydrogen bonding. The bond lengths and angles within two crystallographically independent molecules designated *I* and *II* are presented in Tables II and III.

The coordination around the cobalt atom in both molecules is distorted octahedral. The glycyglycinato ligand is tridentately coordinated through carboxylato oxygen, amino nitrogen and peptide nitrogen atoms, forming two fused, equatorially disposed chelate rings. The diamine ligand is bidentate and forms a five-membered ring. The sixth-coordination position is occupied by the nitro group, *trans* to the N-ethylidene group. The methyl group of the aldehyde residue is in the *syn*-position with respect to the ethylenediamine residue. The occurring of the reaction at the amino group, *trans* to the nitro group is most likely due to the *trans*-influence of the nitro group. Taking into consideration that the amino group of the dipeptide ligand, which is in *cis*-position to the nitro group, reacts with acetaldehyde giving the corresponding Schiff base¹ it seems that steric hindrance of the nitro group is less probable.

As shown in Tables II and III, the geometries of the complex molecules *I* and *II* are essentially the same and in agreement with previously reported data.⁵ The bond lengths and angles in the coordination polyhedra of Co(1) and

TABLE I

Final Fractional Atomic Coordinates and Thermal Parameters with Standard Deviations in Parentheses

Atom	x	y	z	B/Beq ($\times 10^{-4}$ pm ²)
Co(1)	0.5	0.53326(7)	0.5	0.69(2)*
Co(2)	0.03554(18)	-0.03920(7)	0.54454(13)	0.63(2)*
OW(1)	0.3462(10)	0.1589(4)	0.3906(7)	1.3(1)*
OW(2)	-0.1978(11)	0.3251(5)	0.3270(8)	1.6(2)*
O(1)	0.3750(9)	0.5883(4)	0.5787(6)	1.1(3)*
O(2)	0.1100(12)	0.5791(5)	0.5968(7)	3.7(1)
O(3)	0.1708(10)	0.3213(4)	0.3522(7)	3.1(1)
O(4)	0.7923(12)	0.4232(5)	0.6644(7)	3.6(1)
O(5)	0.5488(11)	0.4248(5)	0.6913(7)	3.5(1)
O(6)	-0.0906(9)	-0.0997(4)	0.6188(6)	2.0(1)
O(7)	-0.3720(11)	-0.1048(6)	0.6212(8)	1.8(2)*
O(8)	-0.3059(10)	0.1690(4)	0.3935(6)	2.9(1)
O(9)	0.3150(12)	0.0746(5)	0.7069(7)	3.7(1)
O(10)	0.0678(11)	0.0693(5)	0.7314(7)	3.5(1)
N(1)	0.2708(10)	0.4633(5)	0.4206(7)	0.8(1)*
N(2)	0.5807(12)	0.4622(5)	0.4050(7)	1.1(1)*
N(3)	0.7305(12)	0.6112(5)	0.5856(8)	1.0(1)*
N(4)	0.3888(12)	0.6265(5)	0.3751(8)	1.0(1)*
N(5)	0.6259(12)	0.4496(5)	0.6343(7)	1.1(1)*
N(6)	-0.1974(11)	0.0283(4)	0.4640(7)	0.9(1)*
N(7)	0.1153(11)	0.0349(4)	0.4520(7)	1.0(1)*
N(8)	0.2697(11)	-0.1131(5)	0.6347(7)	0.9(1)*
N(9)	-0.0615(11)	-0.1302(4)	0.4170(6)	1.1(1)*
N(10)	0.1515(11)	0.0451(5)	0.6770(7)	1.0(1)*
C(1)	0.2060(15)	0.5542(6)	0.5500(9)	2.4(1)
C(2)	0.1219(15)	0.4837(6)	0.4507(9)	2.6(2)
C(3)	0.2863(13)	0.3850(5)	0.3801(8)	1.9(1)
C(4)	0.4714(14)	0.3758(6)	0.3736(9)	2.4(1)
C(5)	0.7150(18)	0.6843(7)	0.5034(11)	3.5(2)
C(6)	0.5016(15)	0.7107(7)	0.4247(10)	3.1(2)
C(7)	0.2397(15)	0.6232(6)	0.2662(9)	2.6(2)
C(8)	0.1585(18)	0.6983(7)	0.1750(11)	3.7(2)
C(9)	-0.2670(14)	-0.0706(6)	0.5858(9)	2.4(1)
C(10)	-0.3449(13)	0.0059(6)	0.4954(8)	2.0(1)
C(11)	-0.1862(13)	0.1070(5)	0.4236(8)	1.8(1)
C(12)	-0.0051(13)	0.1185(5)	0.4131(8)	2.1(1)
C(13)	0.2172(15)	-0.2051(6)	0.5888(9)	2.7(2)
C(14)	0.0887(16)	-0.1999(7)	0.4484(10)	3.0(2)
C(15)	-0.2247(15)	-0.1323(6)	0.3144(9)	2.8(2)
C(16)	-0.2895(18)	-0.2027(8)	0.2178(12)	3.7(2)

* To these atoms anisotropic thermal parameters were assigned. The equivalent isotropic thermal parameter is taken as $Beq = 4/3 \sum_i \sum_j \beta_{ij} a_i a_j$

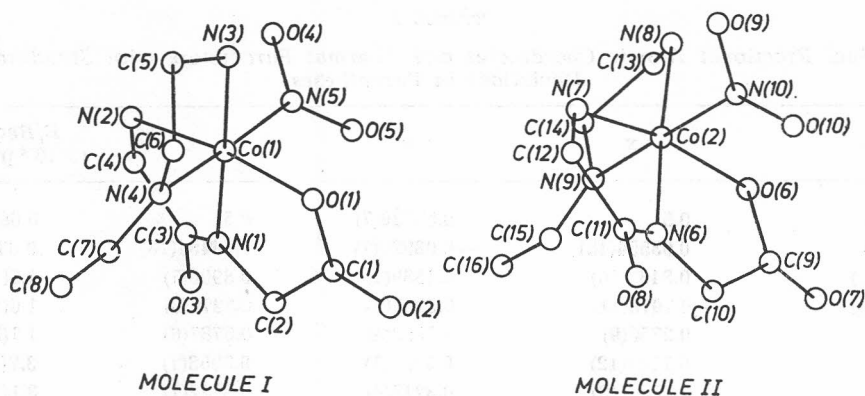


Figure 1. A perspective drawing of the molecules with a numbering scheme of atoms.

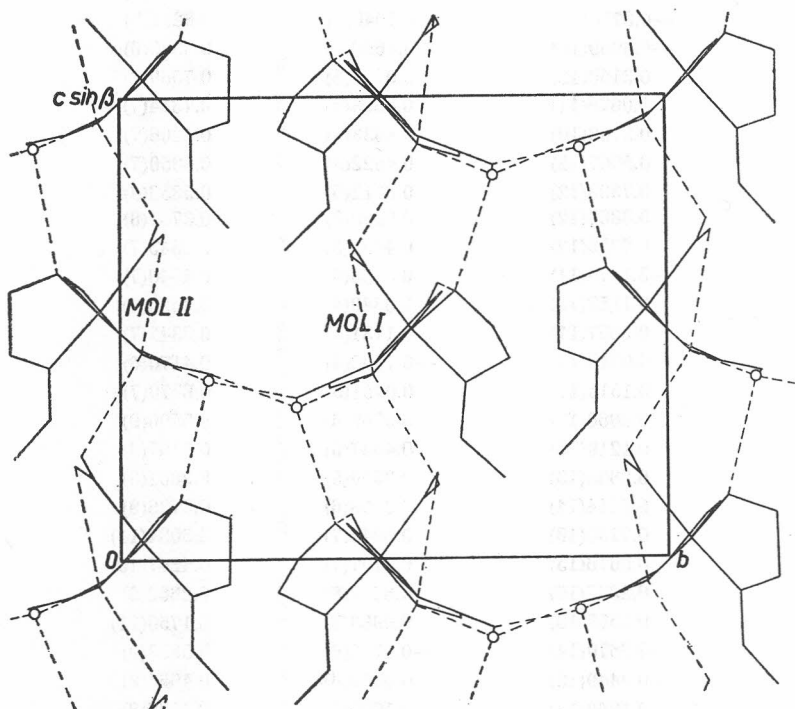


Figure 2. The projection of the structure along a-axis. Hydrogen bonds are presented by broken lines.

Co(2) are within the range of the values found in other complexes of Co(III) with amino acids.⁶ The M—N(peptide) distance, significantly shorter than the M—NH₂ bond length, is observed also in other metal-peptide complexes.^{7,8,9} Bond distances C—N and C—O in the peptide groups of both molecules clearly demonstrate the delocalization of the double bond which occurs due to the

coordination of the dipeptide to the cobalt atom. The usual geometry of ethylenediamine chelate rings is not distorted by formation of the N-ethylidene group; all the bond lengths and angles conform to those already known. The N=C double bond in the N-ethylidene group is also normal.

TABLE II
Bond Lengths (pm) in the Structure of $[\text{Co}(\text{glygly})\text{NO}_2(\text{CH}_3\text{CH}=\text{en})]$

Molecule I		Molecule II	
Co(1) — O(1)	191.1(6)	Co(2) — O(6)	191.3(6)
Co(1) — N(1)	188.1(7)	Co(2) — N(6)	188.1(7)
Co(1) — N(2)	194.4(8)	Co(2) — N(7)	194.1(7)
Co(1) — N(3)	196.3(8)	Co(2) — N(8)	195.0(7)
Co(1) — N(4)	195.6(7)	Co(2) — N(9)	195.2(6)
Co(1) — N(5)	193.2(8)	Co(2) — N(10)	192.1(8)
O(1) — C(1)	129.6(12)	O(6) — C(9)	130.7(11)
O(2) — C(1)	123.3(13)	O(7) — C(9)	123.7(12)
O(3) — C(3)	124.7(10)	O(8) — C(11)	124.5(10)
O(4) — N(5)	123.1(11)	O(9) — N(10)	122.7(11)
O(5) — N(5)	121.4(11)	O(10) — N(10)	122.6(11)
N(1) — C(2)	143.6(12)	N(6) — C(10)	144.8(11)
N(1) — C(3)	132.1(11)	N(6) — C(11)	131.7(10)
N(2) — C(4)	150.4(12)	N(7) — C(12)	150.6(10)
N(3) — C(5)	147.8(14)	N(8) — C(13)	148.4(11)
N(4) — C(6)	148.9(12)	N(9) — C(14)	148.1(13)
N(4) — C(7)	127.1(13)	N(9) — C(15)	127.4(12)
C(1) — C(2)	151.9(13)	C(9) — C(10)	151.7(13)
C(3) — C(4)	150.9(13)	C(11) — C(12)	151.0(12)
C(5) — C(6)	150.2(15)	C(13) — C(14)	152.5(15)
C(7) — C(8)	150.6(15)	C(15) — C(16)	149.7(16)

The conformation of the chelate rings, expressed by puckering parameters,¹⁰ relevant torsion angles and the root mean square torsion angle τ and deviations of the atoms from the least-squares mean planes through the selected group of atoms is presented in Table IV. The analysis of these data shows that the C-terminal five-membered dipeptide rings are remarkably planar, especially the ring in molecule II, with a puckering amplitude of only 1.4 pm, and an average distance of the atoms from their plane of best fit of 0.5 pm. The N-terminal chelates from the cobalt-dipeptide part of the complex are considerably puckered. The angle between the mean planes of the two rings is 14.3 and 11.3° and the maximum displacement from the planes containing all atoms of the cobalt-dipeptide chelates is 22.5 and 18.1 pm in molecules I and II respectively. In the structure of the $[\text{Co}(\text{glygly})(R\text{-ala})\text{NO}_2]^-$ ion ($R\text{-ala} = R\text{-alaninato}$ ligand),⁵ the two chelate rings are almost coplanar, with an angle of 1.5° between mean planes, and the rings are less puckered. The difference in conformation of the dipeptide ligand in molecules I and II of $[\text{Co}(\text{glygly})\text{NO}_2(\text{CH}_3\text{CH}=\text{en})]$ is also evident from the values for the phase angle

TABLE III
Bond Angles ($^{\circ}$) in the Structure of $[\text{Co}(\text{glygly})\text{NO}_2(\text{CH}_3\text{CH}=\text{en})]$

Molecule I		Molecule II	
N(1) — Co(1) — O(1)	84.9(3)	N(6) — Co(2) — O(6)	85.2(3)
N(2) — Co(1) — O(1)	168.5(3)	N(7) — Co(2) — O(6)	168.8(3)
N(3) — Co(1) — O(1)	91.8(3)	N(8) — Co(2) — O(6)	90.8(3)
N(4) — Co(1) — O(1)	88.0(3)	N(9) — Co(2) — O(6)	89.0(3)
N(5) — Co(1) — O(1)	90.1(3)	N(10) — Co(2) — O(6)	91.6(3)
N(2) — Co(1) — N(1)	83.6(3)	N(7) — Co(2) — N(6)	83.6(3)
N(3) — Co(1) — N(1)	176.6(3)	N(8) — Co(2) — N(6)	176.0(3)
N(4) — Co(1) — N(1)	93.8(3)	N(9) — Co(2) — N(6)	94.4(3)
N(5) — Co(1) — N(1)	91.1(3)	N(10) — Co(2) — N(6)	90.3(3)
N(3) — Co(1) — N(2)	99.7(3)	N(8) — Co(2) — N(7)	100.4(3)
N(4) — Co(1) — N(2)	93.3(3)	N(9) — Co(2) — N(7)	91.4(3)
N(5) — Co(1) — N(2)	89.6(3)	N(10) — Co(2) — N(7)	88.9(3)
N(4) — Co(1) — N(3)	85.1(3)	N(9) — Co(2) — N(8)	86.0(3)
N(5) — Co(1) — N(3)	89.9(3)	N(10) — Co(2) — N(8)	89.3(3)
N(5) — Co(1) — N(4)	174.6(3)	N(10) — Co(2) — N(9)	175.2(3)
Co(1) — O(1) — C(1)	114.3(6)	Co(2) — O(6) — C(9)	114.7(6)
Co(1) — N(1) — C(2)	115.5(6)	Co(2) — N(6) — C(10)	115.1(5)
Co(1) — N(1) — C(3)	117.2(6)	Co(2) — N(6) — C(11)	117.4(6)
Co(1) — N(2) — C(4)	108.6(6)	Co(2) — N(7) — C(12)	109.7(5)
Co(1) — N(3) — C(5)	110.2(6)	Co(2) — N(8) — C(13)	108.7(6)
Co(1) — N(4) — C(6)	110.6(6)	Co(2) — N(9) — C(14)	111.2(6)
Co(1) — N(4) — C(7)	128.8(6)	Co(2) — N(9) — C(15)	128.3(6)
Co(1) — N(5) — O(4)	118.1(6)	Co(2) — N(10) — O(9)	117.8(6)
Co(1) — N(5) — O(5)	122.2(6)	Co(2) — N(10) — O(10)	122.1(6)
O(1) — C(1) — C(2)	117.5(8)	O(6) — C(9) — C(10)	116.9(8)
O(1) — C(1) — O(2)	122.7(9)	O(6) — C(9) — O(7)	122.9(9)
O(2) — C(1) — C(2)	119.8(9)	O(7) — C(9) — C(10)	120.2(9)
O(3) — C(3) — C(4)	120.0(8)	O(8) — C(11) — C(12)	119.3(7)
O(3) — C(3) — N(1)	127.2(6)	O(8) — C(11) — N(6)	127.6(8)
O(4) — N(5) — O(5)	119.7(8)	O(9) — N(10) — O(10)	120.1(8)
N(1) — C(2) — C(1)	107.4(8)	N(6) — C(10) — C(9)	108.2(7)
N(1) — C(3) — C(4)	112.8(9)	N(6) — C(11) — C(12)	113.1(7)
N(2) — C(4) — C(3)	110.2(7)	N(7) — C(12) — C(11)	109.9(6)
N(3) — C(5) — C(6)	108.7(9)	N(8) — C(13) — C(14)	106.9(8)
N(4) — C(6) — C(5)	106.4(8)	N(9) — C(14) — C(13)	108.2(8)
N(4) — C(7) — C(8)	126.2(9)	N(9) — C(15) — C(16)	126.1(9)
C(2) — N(1) — C(3)	122.9(7)	C(10) — N(6) — C(11)	122.7(7)
C(6) — N(4) — C(7)	120.7(8)	C(14) — N(9) — C(15)	120.4(8)

TABLE IV
 Conformational parameters of chelate rings*

a) Puckering parameters									
Ring	Atoms			Puckering amplitude q_m (pm)	Phase angle Φ (°)			τ	
1	Co(1), O(1), C(1), C(2), N(1)			5.6	265.4			7.0	
2	Co(1), N(1), C(3), C(4), N(2),			30.5	170.4			19.2	
3	Co(1), N(3), C(5), C(6), N(4)			39.7	94.1			32.1	
4	Co(2), O(6), C(9), C(10), N(6)			1.4	317.9			1.0	
5	Co(2), N(6), C(11), C(12), N(7)			27.3	357.3			17.5	
6	Co(2), N(8), C(13), C(14), N(9)			41.1	72.6			32.5	
b) Selected torsion angles (°)									
O(1) — C(1) — C(2) — N(1)				7.2(11)					
N(1) — C(3) — C(4) — N(2)				-5.0(10)					
N(3) — C(5) — C(6) — N(4)				-47.3(11)					
C(6) — N(4) — C(7) — C(8)				0.3(15)					
O(6) — C(9) — C(10) — N(6)				1.1(11)					
N(6) — C(11) — C(12) — N(7)				0.3(10)					
N(8) — C(13) — C(14) — N(9)				-46.1(11)					
C(14) — N(9) — C(15) — C(16)				2.4(15)					
c) The distance of the atoms from their least squares, mean planes									
Plane	Atoms			Distance (pm)					
1	Rings 1 and 2	Co(1)	-2.1	O(1)	13.5	C(1)	11.0	C(2)	-18.9
		N(1)	-19.9	C(3)	12.8	C(4)	22.5	N(2)	-19.8
2	Rings 4 and 5	Co(2)	-0.6	O(6)	12.8	C(9)	4.8	C(10)	-12.1
		N(6)	-18.2	C(11)	12.7	C(12)	16.6	N(7)	-17.2

* All conformational parameters are calculated for molecules *I* and *II* in position *x*, *y*, *z* from Table I, which have opposite absolute configurations.

Φ (Table IV). It has already been observed that in copper-dipeptide complexes, for which much structural information is available, the chelate ring conformations vary over a wide range, indicating a strong influence of intermolecular contacts.⁸

The two conformers differ also in the conformation of diamine chelate rings. In molecule *I* the conformation is close to twist, with twist axes through the cobalt atom, while in molecule *II* the diamine ring is pure envelope, with the apex at C(13). There is no difference between the conformations of N-ethylidene groups. In both conformers, cobalt and carbon atom bonded to nitrogen are almost coplanar with the atoms of N-ethylidene group.

The average N—O distance of 122 pm and the interbond angle of 120.0° in the coordinated nitro groups agree well with those reported for other Co(III) nitro complexes.^{5,11-13} The least-squares mean plane through the atoms of the nitro group is inclined to the plane of the N-ethylideneethylenediamine ring at an angle of 52.8 and 63.3° in molecules *I* and *II* respectively.

TABLE V
Possible hydrogen bonds

Atom 1	Atom 2	distance (pm)
OW(1)	O(3)	274.1
OW(1)	O(8) ¹	272.9
OW(2)	O(3)	275.8
OW(2)	O(8)	278.4
N(2)	OW(2) ¹	317.6
N(2)	O(4)	286.9
N(2)	O(5) ²	309.0
N(3)	OW(2) ³	295.7
N(3)	O(2) ¹	296.2
N(7)	OW(1)	298.1
N(7)	O(10) ⁴	304.8
N(7)	O(9)	282.4
N(8)	OW(1)	304.2
N(8)	O(7) ¹	291.8

Superscripts refer to atoms in following positions:

- 1 $1 + x, y, z$
- 2 $x, 1 - y, z - 1/2$
- 3 $1 + x, 1 - y, 1/2 + z$
- 4 $x, -y, z - 1/2$

Table V lists the parameters associated with hydrogen bonds that are suggested by geometrical considerations. The molecules of water connect complex molecules *I* and *II* by O—H...O hydrogen bonds in zig-zag chains running parallel to the x-axis. The system of N—H...O hydrogen bonds also participates in connecting the molecules in chains and joins the chains in a three-dimensional network. For the atom N(2) and its analogue N(8) there are two possibilities for one hydrogen bond. It could be that the bond is bifurcated; it is also possible that it is different in the molecules. The latter possibility seems more likely taking into account the shorter O...O distances and different conformations of the corresponding rings.

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SAŽETAK

Kristalna struktura *trans*(NO₂, N=C—CH₃) — (N-etiliden-etilen-diamin)-(glicil-glicinato)-nitro-kobalt(III) monohidrata, $[\text{Co}(\text{glygly})\text{NO}_2(\text{CH}_3\text{CH}=\text{en})]\cdot\text{H}_2\text{O}$

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Kristalna struktura *trans*(NO₂, N=C—CH₃) — (N-etiliden-etilen-diamin)-(glicil-glicinato)-nitro-kobalt(III) monohidrata određena je na osnovi 2519 difraktometrijskih podataka. Kristali su monoklinski, prostorna grupa Pc, a parametri jedinične ćelije $a = 788,4(2)$, $b = 1514,9(2)$, $c = 1220,6(2)$ pm, $\beta = 120,82(4)^\circ$, $M = 339,19$, $Z = 2$, $D_m = 1790$, $D_x = 1800$ kg m⁻³. Struktura je rešena metodom teškog atoma i utaćnjena metodom najmanjih kvadrata primenom pune matrice do faktora pouzdanosti $R = 0,057$. Kristalna struktura sastoji se od enantiomernih parova molekula kompleksa i molekula vode. Asimetričnu jedinicu čine dva kristalografski nezavisna, ali hemijski ekvivalentna molekula kompleksa, koja se razlikuju samo u konformaciji. Koordinacioni poliedar atoma kobalta deformisan je oktaedar. Glicil-glicinato-ligand je tridentatan i koordinovan kobaltu preko karboksilatnog kiseonikovog atoma i azotovih atoma iz amino- i peptidne grupe, gradeći dva kondenzovana helatna prstena, koja zauzimaju ekvatorijalni položaj. Diaminski ligand gradi petočlani prsten. N-etiliden-grupa nalazi se u *trans*-položaju prema nitro-grupi, a metil-grupa aldehidnog ostatka u *syn*-položaju prema etilendiaminskom ostatku.